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IS 8811 (1998): Method for Emission Spectrometric Analysis of Plain Carbon and Low Alloy Steels Point to Plane Technique [MTD 16: Alloy Steels and Forgings]

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"Knowledge is such a treasure which cannot be stolen"

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भारतीय मानक

सादा कार्बन एवं अल्प मिश्रित इस्पात का उत्सर्जन
स्पेक्ट्रोमेट्रिक विश्लेषण प्रणाली प्वाइंट टू प्लेन तकनीक

(पहला पुनरीक्षण)

Indian Standard

METHOD FOR EMISSION SPECTROMETRIC ANALYSIS OF PLAIN CARBON AND LOW ALLOY STEELS POINT TO PLANE TECHNIQUE

(First Revision)

ICS 77.080.20

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Group 3

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard has been prepared with a view to incorporate the latest developments in the field of emission spectrometric analysis.

This standard was first brought out in 1978. The first revision has been undertaken to take into account the experience gained during the period. The method described in this standard is recommended for.analysis of plain carbon and low alloy steels samples of suitable shape and size for all elements for which lines are available in the emission/vacuum emission spectrometer by point to plane spark technique.

The metallurgical state of certain alloys may have some influence on the spectral emission. In these instances, samples and reference materials shall be in the same metallurgical state.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Indian Standard

METHOD FOR EMISSION SPECTROMETRIC ANALYSIS OF PLAIN CARBON AND LOW ALLOY STEELS POINT TO PLANE TECHNIQUE

(First Revision)

1 SCOPE

1.1 This standard gives a generalised spectrometric analysis procedure for determining the following elements when present as alloying element or as impurity in plain carbon and low alloy steels with a minimum iron content of 94 percent. The possible analytical and internal standard lines alongwith the concentration ranges are also given below for .information:

Element	*Analytical Line* (nm)	*Internal Standard Line* (nm)	*Concentration Range* (percent by mass)
Aluminium	394.4 308.2	271.44	0.005 to 0.15
Arsenic	197.20 193.76	271.44	0.005 to 0.05
Boron	182.64 182.59	271.44	0.005 to 0.010
Carbon	193.09	271.44	0.01 to 1.5
Chromium	298.92 267.72	271.44 271.44	0.05 to 5.0
Cobalt	345.35 228.62	271.44	0.01 to 0.20
Copper	327.40 213.6	271.44	0.01 to 0.5
Manganese	293.31 255.86	271.44	0.01 to 2.0
Molybdenum	379.83 277.54 386.41	271.44	0.01 to 1.5
Nickel	288.16 251.61	271.44	0.05 to 5.0
Niobium	319.5	271.44	0.008 to 0.076
Phosphorus	178.29	271.44	0.005 to 0.10
Silicon	288.16 51.61	271.44	0.05 to 2.0
Sulphur	180.73	271.44	0.005 to 0.10
Tin	189.99	271.44	0.003 to 0.10
Titanium	337.28	271.44	0.01 to 0.25
Vanadium	310.23 311.07	271.44	0.01 to 1.0
Zirconium	343.82	271.44	0.001 to 0.05

1.2 More elements and varied detection limits can be achieved by choosing suitable lines while selecting the spectrometer.

2 REFERENCE

2.1 The following Indian Standard is a necessary adjunct to this standard:

IS No.	Title
7072:1973	Glossary of terms relating to emission spectroscopy

3 SUMMARY OF METHOD

3.1 The sample is excited by controlled spark/discharge, using a point (counter electrode) to plane (polished flat surface of sample). The radiation from spark/discharge, is dispersed by a quartz prism/grating. The radiant energy (intensity) of the selected analytical lines are converted to electrical impulses by photomultiplier tube, which is amplified and stored in capacitor. The charge from each capacitor is measured and converted into intensity ratios. The concentrations of elements are calculated from analytical working curves prepared by exposing standard samples of similar type.

4 SPECTROMETER AND GENERAL REQUIREMENTS

4.1 Any make of spectrometer having good reciprocal linear dispersion and good resolution is recommended. The sensitive lines of certain elements like carbon, sulphur, phosphorus, arsenic, boron, etc, are in vacuum ultra violet region. To avoid absorption of radiation of these lines, a vacuum type spectrometer is most suitable.

4.2 The spectrometer should have a wave length coverage of 1 700 to 4 000A° for analytical lines.

4.3 Focal length, grating parameters, reciprocal linear dispersion, primary slit width, secondary slit width and wave length coverage shall be as per manufacturer's discretion.

4.4 The lines selected for each element shall be of proven satisfactory reproducibility. Though, most of the lines are common, the lines shall be selected by the instrument manufacturer to get maximum optical output with minimum interference.

4.5 The spectrometer shall be kept in the following laboratory environments:

Temperature	19°C to 25°C
Relative humidity	45 to 60 percent
Atmosphere	Dust-free

4.6 The electric power tolerance shall be ± 5 percent preferably ± 2 percent and filtered to prevent radio frequency interference, with good r.f. grounding system when working with microprocessors and computers.

4.7 If the spectrometer is provided with vacuum system, it shall be the manufacturer's choice.

NOTES

1 The manner and frequency of checking the optical alignments and electrical parameters will depend on factors such as type of spectrometer, manufacturer, the variety of analytical problems encountered and frequency of use. For details of setting and operating a particular spectrometer, refer to the manufacturer's handbook. Each laboratory should establish a suitable check procedure.

2 Instrument parameters may vary with each instrument. However, the parameters given in Annex A have been used successfully in several laboratories and are given for guidance.

5 ELECTRODES

5.1 The counter electrode shall be of pure silver or thoriated tungsten.

5.2 The shape and angle of upper tip shall be as per the requirement of the instrument.

5.3 The electrode and tip shall be kept clean.

5.4 The spark gap between electrode and specimen shall be as per the manufacturer's instruction manual.

6 SPARK STAND AND ARGON SYSTEM

6.1 Spark stand shall be mounted directly on the spectrometer, and equipped to hold flat specimens and counter electrode.

6.2 Argon flow connection during flushing and sparking with automatic controls shall be provided on the spark stand.

6.3 Water cooling of the stand will be advantageous.

6.4 Argon shall be of purity 99.995 percent minimum. A reducing atmospheric furnace at 425/450°C with Mg/Ti molecular sieves shall be used to remove oxygen/moisture traces from argon.

NOTE — If Argon of this purity is not available it can be purified by passing through a reducing atmospheric furnace at 425/450°C with Mg/Ti and further over molecular sieves.

6.5 Argon pressure and flow rate shall be as per manufacturer's instruction.

7 EXCITATION SOURCE

7.1 For precision and accuracy of analysis, a triggered/controlled high voltage capacitor discharge shall be used.

7.2 The excitation parameters depend mainly on the nature of samples to be analysed, elements and the concentration levels to be determinated. These can be achieved by different combinations of capacitance, potential (voltage), resistance and inductance. Each manufacturer recommends special combinations. However, source parameters used successfully by several laboratories are given in Annex B and can be used as guidance.

7.3 The source parameters shall also include peak current, current pulse duration and number of discharges per second.

7.4 The established parameters should be available with each spectrometer.

7.5 Pre-burn period, exposure period and exposure conditions shall be established by the user depending upon the type of samples being analysed and as per the recommendations of the manufacturer. However, the parameters given in Annex C were sucessfully used in several laboratories and thus can be used as guidance.

8 MEASURING SYSTEM

8.1 The spectrometer measuring system consists of photo-multiplier tubes with individual voltage adjustments, amplifiers, capacitors to store the output voltage and system to measure the capacitor charges and programmes for the sequence of operations.

8.2 For measuring the capacitor charge, different systems are used and shall be as per the recommendations of the manufacturer.

9 SAMPLING AND SAMPLE PREPARATION

9.1 Sample for spectrometer analysis shall be of approximate 30 to 40 mm diameter and 10 to 20 mm thickness. Smaller size is not recommended due to heating up during excitation and spark discharge.

9.2 Sample produced shall be homogeneous and free from porosity, voids and inclusions.

9.3 Any of the following methods shall be used for liquid steel sampling:

a) Pouring into cast-iron mould,

b) Pouring into copper moulds (single or split type), and

c) Immersion or lollypop sampling.

The moulds may also be of water-cooled type.

9.4 Normally, liquid steel samples shall be Al/Ti or Zr 'Killed'. The amount of deoxidant to be used depends on the metal condition.

9.5 Other samples can be of forged or rolled products.

9.6 The sample preparation consists of cutting (if required), rough grinding, and final finishing with 60/120 grit paper.

9.7 The prepared sample shall have a smooth flat uniformly finished surface.

NOTE — The method of sampling and finishing is of individual choice and depends upon the product range.

10 STANDARD SAMPLES

10.1 Standard samples shall be of two types:

a) *Primary Standards* — preferably from qualified agencies engaged in preparation of spectrographic standards.

b) *Working Standards* — These are prepared by the user depending upon his product range and type of composition.

11 CALIBRATION AND STANDARDIZATION

11.1 The spectrometer shall be stabilised as per the manufacturer's instruction, and all the parameters regarding profiling, vacuum, argon flow, excitation conditions are set to the required level.

11.2 The internal standard method shall be applied for measuring the intensity or radiant energy of different lines and for making working curves. In this method, the discharge is terminated at a predetermined accumulated level of intensity for internal standard iron line.

11.3 Using low and high alloy composition standards set the dynode/attenuater to control the photomultiplier tube, to get the required voltage output for each line for minimum and maximum percentage of the element.

11.4 Excite standard samples with varying elemental percentages several times (to take care of the short term drift) and measure the intensity readings.

11.5 The working curve shall be prepared as in **11.2**, using intensity ratios *vs* concentration ratios.

11.6 Depending upon the user programme, wider concentration ranges can be plotted in segments with expanded scales.

11.7 While preparing curves the following points have to be taken into account:

a) Background correction,

b) Inter-elemental interference,

c) Metallurgical condition/state, and

d) Analytical curve shift/rotation.

In most of the latest version of spectrometers, the software supplied by the manufacturer calculates the correction for the above factors. These are explained briefly in **11.7.1** to **11.7.4**.

11.7.1 Line-to-background correction has to be applied for each channel/line to get more accurate analysis, especially in lower concentration levels. This is normally done by subtracting line background reading from the actual intensity output. The detection limit is also enhanced by dynamic background correction.

11.7.2 In many cases, interferences from other elements are significant, especially at lower

concentration levels. This may lead to suppression or enhancement of intensity. Interference factor is established by exposing different standards of varying matrix composition.

11.7.3 Corrections for metallurgical state/condition are applied as per users choice of samples. This is normally achieved by selection of working standards of similar metallurgical condition as that of samples to be analysed. The working curve prepared from primary standard are corrected by exposing the working standards. These are also known as type standardization.

11.7.4 Analytical curve shift/rotation is caused by the dirty optics, change in optics, excitation source, line voltage, spectrometer electronics and ambient room conditions. Excite a set of standards, and taken intensity readings and correct the curves for drift.

11.8 Accuracy of standardization depends mainly on the long-term stability of the emission spectrometer. If the values are within the standard deviations of the standard samples, no correction need be applied. If there is considerably deviation, recalibrate and reestablish the curve.

11.9 The instrument and calibration shall be evaluated periodically and standard errors are corrected, so as to enable the operator to get accurate results.

12 PROCEDURE

12.1 Set-up the spectometer as per the standard procedure, clean the excitation stand and electrode.

12.2 Prepare the sample as per **9**.

12.3 Excite the sample and take intensity readings in duplicate or triplicate.

12.4 From the average intensity reading calculate the concentration of different elements from the working curve.

12.5 Conversion of scale, digital voltmeter or print-out readings into element percentages, coupled with evaluation of correction factors, may also be carried out through computers and programmable calculators, which are previously programmed with correction and calibration data. Each curve is stored in memory. A linear arithmetic formula or polynomials of second or higher orders have been used to represent analytical curves in the memory register of the automatic devices. Conversion of intensity ratio into concentration is done automatically.

12.6 Latest model spectrometers are equipped with visual display units and printouts for obtaining results.

ANNEX A

(*Clause* 4.7, *Note*)

INSTRUMENT PARAMETERS

Focal length	0.75 to 1.0 m
Wave length coverage	1 700 to 4 000 A°
Reciprocal linear dispersion	6 A° mm *Min*
Vacuum	25 µm or below
Primary slit width	20 µm to 50 µm
Secondary slit width	30 µm to 200 µm

ANNEX B

(*Clause* 7.2)

SOURCE PARAMETERS

Capacitance, µF	10 to 15
Inductance, µH	50 to 70
Resistance	3 to 5
Potential, V	940 to 1 000
r.f. current, A	0.3 to 0.8
Discharge/sec	60

Once parameters (selected from this) are established, maintain them carefully.

ANNEX C

(*Clause* 7.5)

EXPOSURE CONDITIONS

Pre-flush period	5 to 15 sec
Pre-burn period	5 to 20 sec
Exposure period	3 to 30 sec
Argon flow flush period	2.5 to 25 *l*/min
Argon flow pre-burn	2.5 to 25 *l*/min
Argon flow exposure	2.5 to 15 *l*/min
Electrode angle	90° to 120°
Spark gap	3 to 5 mm

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